



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

# 20  
7.3.2d

APPLICANT: GUIDOTTI ET AL. )

SERIAL NUMBER: 09/315,251 )

Group-Art Unit: 174

FILED: May 20, 1999 )

FOR: ENERGY STORAGE AND )  
CONVERSION DEVICES USING )  
THERMAL SPRAYED )  
ELECTRODES )

Examiner: J. Maple

DECLARATION PURSUANT TO 37 C.F.R. 1.132

The Assistant Commissioner of Patents  
Washington, D.C. 20231

Sir:

RECEIVED  
JUL 01 2002  
TC 1700

Tongsan D. Xiao, declares and says that:

1. I have worked in the field of thermally sprayed coatings for 10 years. I  
an inventor of the above referenced patent application.

2. I have read, understood, and am familiar with the above-referenced  
application. I have also read, understood, and am familiar with the U.S. Patent No.  
5,716,422 to Muffoletto et al.

3. During initial investigations into thermally spraying active materials the  
thermally unstable at thermal spray temperatures, attempts were made to form an FeS  
(pyrite) coating by thermally spraying FeS<sub>2</sub> as described by Muffoletto. First attempt:

employed high-velocity oxygen flame (HVOF). These attempts were unsuccessful in that  $\text{FeS}_2$  was not deposited. The deposited material was analyzed by X-ray diffraction (XRD) and was found to be primarily  $\text{Fe}_2\text{O}_3$ . Further attempts to obtain an  $\text{FeS}_2$  coating employed dc-arc plasma spray under an argon cover (250 SCFH) with arc currents of between 200 and 300 A. XRD of the deposited material indicated  $\text{Fe}_7\text{S}_8$  and not  $\text{FeS}_2$ . Representative data is shown in Fig. 1 attached hereto.

4.  $\text{FeS}_2$  was plasma sprayed with elemental sulfur. The primary phase of the resulting deposit (>95%) was  $\text{FeS}_2$  as shown by the X-ray data in Figure 1.

5. Discharge traces were recorded for cells employing natural  $\text{FeS}_2$ ,  $\text{FeS}_2$  plasma sprayed without sulfur and  $\text{FeS}_2$  plasma sprayed with sulfur. These discharge traces are shown in Fig. 2 attached hereto. The discharge traces corroborate the above described XRD data in that the  $\text{FeS}_2$  plasma sprayed without sulfur exhibits a significantly different trace than the natural pyrite and the  $\text{FeS}_2$  plasma sprayed with sulfur. Additionally the discharge traces of the cell employing  $\text{FeS}_2$  plasma sprayed with sulfur paralleled the discharge traces of the cell employing natural pyrite.

6. Thus, based on my knowledge of the thermal spray field and the above described experiments and data, I believe the presence of a protective coating such as elemental sulfur is to be necessary to produce a thermally sprayed electrode comprising  $\text{FeS}_2$  or other material that is unstable at thermal spray temperatures.

7. I declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and those like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or document or any patent resulting therefrom.

Date: 6/26/02

Tongsan D. Xiao  
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